Summary:

The primary activities over the last week were information gathering, following up on leads provided by the consultants and developing a plan for removal of the corrosive material in the storage tanks

Ronnie followed up with Bill Gumprecht (Cecon Consulting) and Bradford Boyce (??). He got further confirmation that it will be necessary to continuously remove the tars (and other heavy components) generated in the TCE + HF to 133A reaction. The resulting waste stream will also contain the catalyst. Ronnie will look at options for separating the catalyst from the tar/heavy stream in an additional step in Norphlet's process. If it isn't feasible to do it on site, the waste stream will need to be sent to an outside facility for processing Through a contact provided by Bill, Ronnie found a hazardous waste facility in Colorado that can recover and regenerate antimony based catalysts from the tars/heavies and dispose of the residual organic waste They will evaluate whether they can provide the same services for tantalum

In a phone conversation with Steve Owens, Ronnie found out that Iikubo (the 133A consultant we were trying to contact last week) is still doing consulting work and is currently in Asia Steve has worked with Iikubo on other projects and, as a result of the relationship, may be able to help us in getting Iikubo to work with us. Iikubo would likely be extremely helpful in assisting us with our catalyst selection, tar removal system and other issues associated with the 133A process

Vic Forte has come up with a detailed plan for processing the corrosive waste material in the storage tanks The lighter components that we recover will be routed to another storage tank The remaining tars and organics will be put in lined 55 gal drums for analysis and later disposal. If all goes as planned, the corrosion concerns will be eliminated and the volume of hazardous waste will be significantly reduced.

Process Issues to Overcome - Updates

Stage 1 Reaction (TCE + HF to 133A)

We have 2 key decisions to make regarding the Stage 1 Reaction:

1. How to design the Tars/Heavies removal system
2. Which catalyst to use (TaF_5 or $SbCl_5$)

Although the consultant's we've been in contact with so far have provided a wealth of information and some general direction, none have specific experience with or recommendations regarding design or operating conditions We would benefit greatly from some consulting assistance from Iikubo as he was associated with the plant that produced 133A for DuPont In a phone conversation with Steve Owens last week, Ronnie learned that Iikubo is still working as a consultant and that he is currently in Asia Apparently our contact information for Iikubo was out of date, which would explain why he hadn't returned our e-mails and phone calls. Steve and Iikubo have worked together on other projects in the past and Steve gave Ronnie the indication that Iikubo might we willing to help on our project Whether the consulting relationship would be with Iikubo directly or through Steve Owens is unknown at this point

If we switch to $SbCl_5$, we may need to use a different type of reactor Bill Gumprecht offered to send Ronnie Jackson design information on Antimony reactors

Tars/Heavies Removal:

We got additional confirmation that a Tars/Heavies removal system would be required for the Stage 1 reaction. Bradford Boyce, in a conference call on Friday, 2/8, said that heavies and tars were an inherent part of the reaction and would need to be removed from the system. An unavoidable consequence of removing the tars/heavies from the process is that the catalyst will be removed along with them He agreed that a distillation column would be required to separate the recyclable components from the Tar/Catalyst stream and speculated that a tantalum based catalyst could be recovered via an HF wash and subsequent phase separation If we use an Antimony based catalyst or are unable to recover the Tantalum catalyst in our process, we will need to send the Tar/Catalyst waste to an off-site contractor for catalyst recovery and waste disposal

TaF_5 vs. $SBCl_5$ Catalyst:

In the phone conversation with Bradford, he commented on the relative advantages and disadvantages of the two catalysts He re-iterated what we knew previously but brought up a couple of new concerns regarding the use of Antimony; 1) that $SbCl_5$ was volatile and could end up in places where we wouldn't want it and, 2) that the chlorine co-feed required for use with Antimony needed to be carefully controlled to prevent corrosion of downstream carbon steel equipment He admitted that antimony has been used in similar processes for several years in carbon steel equipment but wanted us to be aware of the corrosion potential

In regards to the form of Tantalum to use as the starting material for TaF_5, Ronnie asked Bradford if Tantalum Powder or Tantalum Pentachloride ($TaCl_5$) should be used (Bill Gumprecht had previously recommended $TaCl_5$). Bradford said that the decision was purely an economic one, whichever was cheaper, on a molar basis, should be used.

Tars Disposal/Catalyst Recovery

We had a conference call on Friday with Dick Angstadt of Chemical and Metal Industries, Hudson, CO Bill Gumprecht had previously provided Ronnie with the contact information. Dick's company provides hazardous waste disposal and Antimony recovery services They separate the organics from the antimony, ship the organics to a licensed incineration facility and recover and regenerate the Antimony Catalyst for return shipment to the customer Dick confirmed that he could provide these services to Norphlet if we switch to the $SbCl_5$ catalyst Dick also said that his company is interested in expanding their range of services and would welcome the opportunity to evaluate the feasibility of recovering Tantalum from an organics stream and Chromium from carbon based catalysts (as in Norphlet's catalyst for the Stage 2 reaction) We agreed to send Dick a sample of the Tars/catalyst for his evaluation Ronnie asked about other potential hazardous materials incineration facilities Dick mentioned that Clean Harbors, located in El Dorado, AR, provided incineration services. He wasn't sure if they had the capability to process Fluoride containing wastes; apparently very few facilities can.

Stage 2 Reaction (133A to 134A):

In his phone conversation with Steve Owens, Ronnie was told that there may be a "2nd Generation" catalyst for the reaction. It is apparently a Chromium Gel No other specifics were offered.

Patent Issues

Evert met with Judd Hammond on Monday, 2/4 to follow up on my concern about running the Stage 2 reaction (133A to 134A) in the temperature range cited in Patent '276 and our potential switch from Tantalum Pentafluoride (TaF_5) to Antimony Pentachloride ($SbCl_5$) as a catalyst for the Stage 1 reaction (TCE + HF to 133A) Judd advised us that the 2 reaction steps must be considered together and that as long as we remain below 200 ^{0}C in the Stage 1 Reaction (as the Norphlet process is designed), we will not be infringing on the patent In regards to Antimony as a catalyst, Judd's research indicates that the technology has been around for decades and that the original patents have expired.

Additional Equipment/Supplies/Materials Needed

At this point, we are certain that a tar (and "heavies") removal system will be required for the Stage 1 reaction This system will include a distillation column to separate the lighter recyclable components from the tars and catalyst and possibly an additional (yet to be defined) process step to recover the catalyst from the tars It is also likely that a Stripper will be required to achieve the required purity in the HCl stream and that a water wash step will be needed for HF removal from the 134A These items will largely determine the magnitude of additional capital spending required and will have significant lead times In order to be in a position to specify and order these items, the process modeling and simulation must be completed. Ronnie Jackson is planning to meet with Steve Owens next week in an attempt to expedite this work In the meantime, Ronnie, Vic Forte and I will work on an equipment specification, ordering and installation plan to keep the project schedule as short as possible.

Accumulated Off-Spec Inventory:

Vic has developed a detailed plan where the re-usable components will be recovered in one of the product storage tanks and the tars and catalysts will be captured in lined 55 gal lined drums Once this process is completed, the tank corrosion concerns will be eliminated and we will have adequate time to analyze the waste material and locate the most economical disposal option. The proposed plan has been shared with Bill Gumprecht and Bradford Boyce and both agree that it is a good approach The drums and liners are readily available Once Vic has approval to proceed, he will order the drums and carry out the separation.

Site Environmental Issues:

Engineering/Procurement/Construction (EPC) Firm Evaluations:

These evaluations will be scheduled once we have more definition on the scope of the process changes.

Plans for week of February 11th

I don't have plans to travel to Norphlet this week (subject to change) but will be in regular contact with Vic, Ronnie and Evert by phone

Open Issues:

- Implement plan for removal of corrosive material from storage tanks
- Locate Process Consultant for TCE/HF to R-133A reaction (Iikubo)
- TaF_5 vs $SbCl_5$ decision
- Process Engineering – define additional process steps/changes
- Run Process Simulation to identify flow, equipment and utility requirements (Steve Owens)
- Develop Project Schedule (Design/Equipment Ordering/Installation)
- Visit Engineering/Construction firms to assess capabilities for completing plant modifications/additions